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## PATENT SPECIFICATION

NO DRAWINGS

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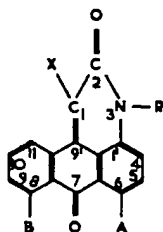
## COMPLETE SPECIFICATION

## Colouring Process

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, of Imperial Chemical House, Millbank, London, S.W.1., a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a colouring process and more particularly relates to a process for the mass-colouration of polypropylene.

According to the invention there is provided a process for the mass-colouration of polypropylene using as the colouring matter a pigment of the formula:—



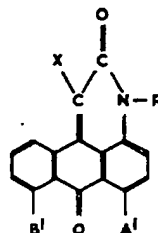
wherein R represents a hydrogen atom or an alkyl or cycloalkyl radical; X represents a hydrogen atom or an acetyl or carboalkoxy group; and one of A and B represents a hydrogen atom and the other of A and B represents an anthraquinonylamino, anthrapyridonylamino or anthrapyridonyl radical.

The alkyl radicals represented by R are preferably lower alkyl radicals containing from 1 to 4 carbon atoms such as ethyl, propyl, butyl and, above all, methyl radicals. As an example of a cyclo-alkyl radical represented by R there may be mentioned the cyclo-hexyl radical. It is however preferred that R represents the methyl radical.

The carboalkoxy groups represented by X are preferably carboalkoxy groups wherein the alkoxy radical contains from 1 to 4 carbon atoms, and as examples of such groups there may be mentioned carbomethoxy, carboethoxy, [Price 4s. 6d.]

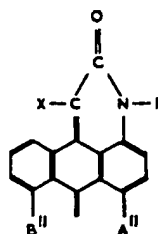
carbopropoxy and carbobutoxy groups. It is however preferred that X represents a hydrogen atom.

The pigments used in the process of the invention wherein one of A and B is an anthraquinonylamino or anthrapyridonylamino radical may themselves be obtained by reacting an aminoanthrapyridone of the formula:



wherein X and R have the meanings stated above, and one of A' and B' represents a hydrogen atom and the other of A' and B' represents an amino group, with a halogenoanthraquinone or halogenoanthrapyridone respectively, in a solvent such as nitrobenzene and in the presence of an acid-binding agent and a copper catalyst; for example as described in British Specification No. 13686 of 1907.

The pigments used in the process of the invention wherein one of A and B is an anthrapyridonyl radical may themselves be obtained by heating a halogenoanthrapyridone of the formula:—



wherein X and R have the meanings stated above, and one of A' and B' represents a hydrogen atom and the other represents a halogen atom, in a solvent such as dimethylformamide or nitrobenzene in the presence of an acid-binding agent and a copper catalyst.

The process of the invention can be conveniently brought about by mixing the pigment, in finely divided form, with the solid polypropylene (such as polypropylene in the form of powder or small granules) and thereafter melting and spinning the so-coated polypropylene by known techniques. When the finely divided pigment is in the form of an aqueous dispersion then, after mixing the said aqueous dispersion with the polypropylene, it is preferred to dry the mixture before melt spinning the product.

It is preferred that the so-coated polypropylene after melting is spun into fibres. The polypropylene fibre so obtained is coloured in yellow to violet shades which possess excellent fastness to light, to wet treatments such as washing and to dry heat treatments. The pigments used in the process of the invention are tinctorially strong and are not degraded by the high temperature used in the melt spinning operations so that, by the process of the invention, it is possible to also obtain polypropylene fibres which are coloured in bright deep shades.

The invention is illustrated but not limited by the following Examples in which the parts and percentages are by weight.

#### EXAMPLE 1.

1 part of 8:8'-bis-(3-methyl-1'':9''-anthrapyridonyl)amine in finely divided form is intimately mixed with 100 parts of powdered polypropylene. The resulting mixture is passed through a heated screw extruder so as to form a compact rod which is then cut into small pieces. The resulting pieces of pigmented polypropylene are then melt spun to form fibres of a deep red shade which possess excellent fastness to light, heat, and to steaming, washing, dry cleaning and rubbing treatments.

In the above Example the stage of passing the polypropylene through a heated screw extruder can be replaced by hot pressing the polypropylene into a solid rod or alternatively the stages of passing the polypropylene through a heated screw extruder and then melt spinning the polypropylene can be combined into one stage in a suitable apparatus, when similar results are obtained.

If in place of the 1 part of 8:8'-bis(3-methyl-1'':9''-anthrapyridonyl)amine used in the above Example there are used 1 part of the corresponding 6:8'- or 6:6'-isomers, fibres of red or deep violet shades are obtained.

The 8:8' - bis(3 - methyl - 1'':9'' - anthrapyridonyl)amine used in the above Example may be obtained by reacting 8-amino-3-methyl - 1':9' - anthrapyridone (which is

described in British Specification No. 13686 of 1907) with 8-chloro-3-methyl-1':9'-anthrapyridone in nitrobenzene in the presence of an acid-binding agent and a copper catalyst. The resulting product separates out from the reaction mixture and is filtered off, washed with benzene, then with a dilute aqueous solution of hydrochloric acid, and dried. The product is converted to a finely divided form by any of the processes known for converting pigments to a fine state of subdivision, for example by milling or acid-pasting.

The corresponding 6:8'-isomer was prepared in a similar fashion from 6-amino-3-methyl-1':9'-anthrapyridone; while the 6:6'-isomer is described in British Specification No. 13686 of 1907.

#### EXAMPLE 2.

10 parts of an aqueous dispersion containing 10% of 6-(anthraquinon-2''-ylamino)-3-methyl-1':9'-anthrapyridone is mixed with 100 parts of powdered polypropylene and the resulting mixture is dried in warm air. The dry mixture is then compacted, for example by passage through a heated screw extruder, and is then melt spun to yield bluish-red fibres of excellent fastness to light and to hot pressing, steaming, dry cleaning, washing and to rubbing.

The 6-(anthraquinon-2''-ylamino)-3-methyl-1':9'-anthrapyridone was prepared as described in British Specification No. 13686 of 1907, and then converted to a 10% aqueous dispersion by known methods.

In place of the 10 parts of the 10% aqueous dispersion of 6-(anthraquinon-2''-ylamino)-3-methyl-1':9'-anthrapyridone used in the above Example there are used 10 parts of 10% aqueous dispersions of 6-(anthraquinon-1''-ylamino)-3-methyl-1':9'-anthrapyridone or 8-(anthraquinon - 1' ylamino) - 3 - methyl-1':9'-anthrapyridone whereby the polypropylene fibres are coloured in purple or brownish-red shades respectively.

The first of these pigments was obtained as described in British Specification No. 13686 of 1907 while the second was prepared in a similar manner from 8-chloro-3-methyl-1':9'-anthrapyridone and 1-aminoanthraquinone.

#### EXAMPLE 3.

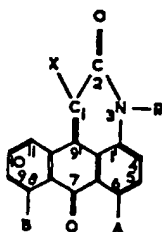
1 part of bis(3-methyl-1':9'-anthrapyridon-6-yl) in finely divided form is mixed with 100 parts of powdered polypropylene, and the resulting mixture is then compacted and melt spun to yield fibres of a deep yellow shade which have excellent fastness to light and to hot pressing, steaming, washing, dry cleaning and rubbing.

The bis(3-methyl-1':9'-anthrapyridon-6-yl) used in the above Example was obtained by heating a mixture of 34 parts of 6-bromo-3-methyl-1':9'-anthrapyridone, 17 parts of copper bronze and 285 parts of dimethylform-

amide for 15 hours at 150° C. The precipitated solid was then filtered off, washed with dimethylformamide, then with water, treated with a dilute aqueous solution of nitric acid and finally washed with water. The product was then dried and subsequently converted to a finely divided form by known techniques.

WHAT WE CLAIM IS:—

1. Process for the mass-coloration of polypropylene which comprises using as the colouring matter a pigment of the formula:—



wherein R represents a hydrogen atom or an alkyl or cycloalkyl radical; X represents a hydrogen atom or an acetyl or carboalkoxy group; and one of A and B represents a hydrogen atom and the other of A and B represents an anthraquinonylamino, anthrapyridonylamino or anthrapyridonyl radical.

2. Process as claimed in Claim 1 wherein the pigment is mixed with the solid polypropylene, and the resulting mixture is melted and spun into fibres.

3. Process for the mass-coloration of polypropylene as hereinbefore particularly described especially with reference to any of the Examples.

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Agent for the Applicants.